

SINTESIS, KARAKTERISASI DAN AKTIVITAS KATALIS

$\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$ PADA REAKSI TRIMETILHIDROKUINON DAN ISOFITOL

Oleh :

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Dosen Pembimbing :

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PENDAHULUAN

Katalis



PENDAHULUAN

TMHQ

+

Isofitol



Produk

Katalis

PENDAHULUAN

Katalis

Katalis + Homogen

H_2SO_4 , HCl , BF_3 , AlCl_3 dan FeCl_2/HCl
(Bonrath & Netscher, 2005)

- Sulit dipisahkan
- Korosif
- Katalis hanya bisa dipakai sekali (Lien, 2012)

Katalis Heterogen

Nafion/ SiO_2 (Laufer dkk., 2005), $\text{M}(\text{OTf})_3$
(Schrager & Bonrath, 2007)

- Mudah dipisahkan
- Tidak Korosif
- Katalis bisa dipakai kembali (Nguyen dkk., 2012)

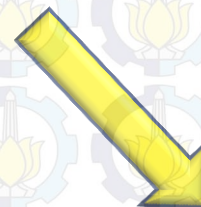
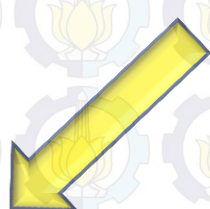
PENDAHULUAN

Keasaman Katalis

Asam Lewis

Asam Brønsted

Asam Lewis dan
Brønsted



RUMUSAN MASALAH

Katalis
 MgF_2

Keasaman <<

Modifikasi Keasaman

Brønsted

Penambahan OH

Lewis

Doping Zn



Keasaman

Konversi

Yield



TUJUAN PENELITIAN



Aktivitas



TMHQ + Isofitol

* X = 0 ; 0,025 ; 0,05 ; 0,075 ; 0,1 ; 0,15

Aplikasi pada reaksi TMHQ dan Isofitol

Manfaat



Karakterisasi

- XRD
- FTIR
- Adsorpsi Piridin-FTIR
- Adsorpsi gas N_2

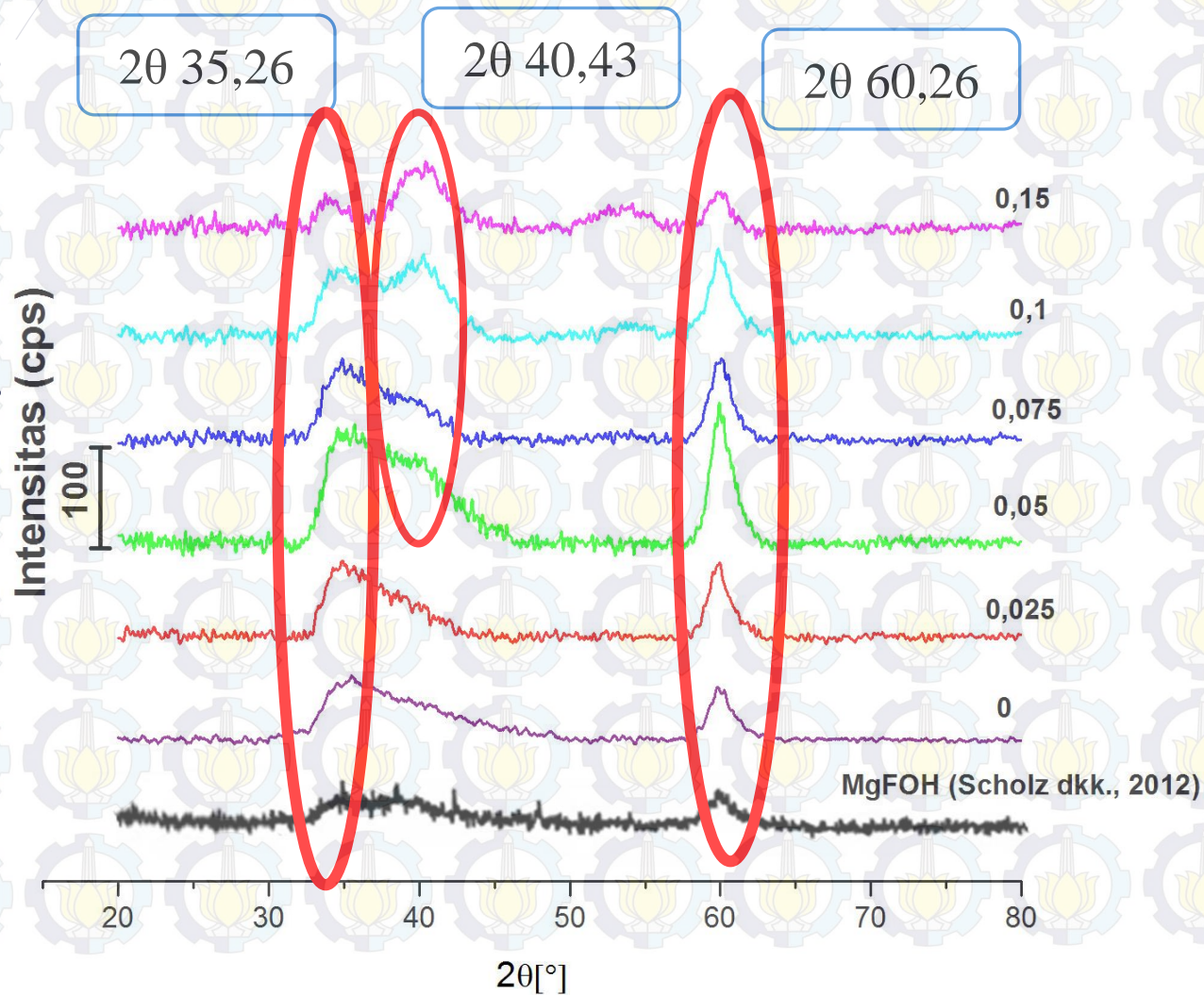
Uji Katalisis

TMHQ + Isofitol

↓
Produk
↓
UV-Vis

* $x = 0 ; 0,025 ; 0,05 ; 0,075 ; 0,10$ dan $0,15$

Difraktogram $\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$



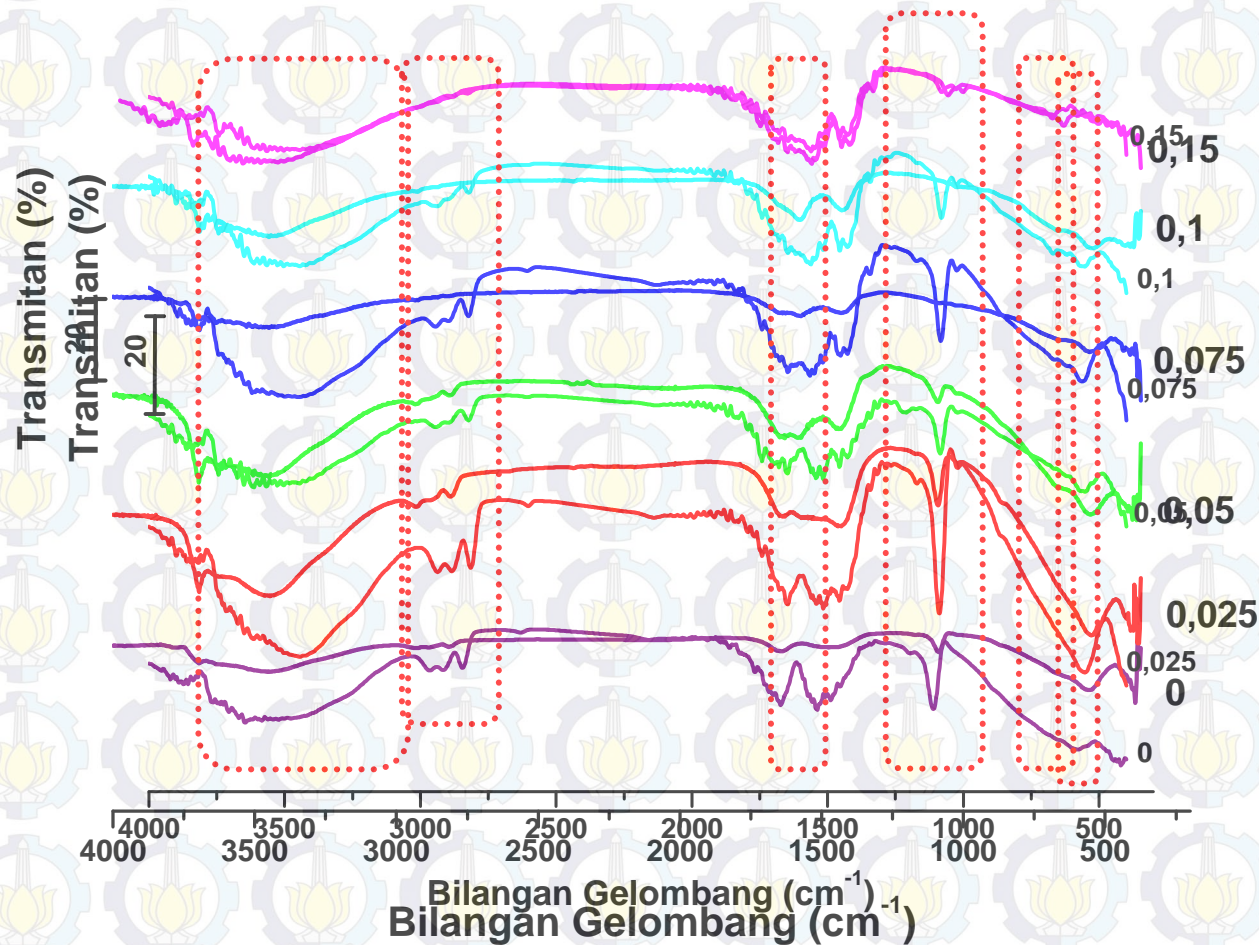
Tabel pergeseran 2 θ pada katalis $\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$

Nilai x pada katalis $\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$	Puncak 1		Puncak 2		Puncak 3	
	2 θ (°)	Int. (cps)	2 θ (°)	Int. (cps)	2 θ (°)	Int. (cps)
0	35,26	141	-	-	60,26	108
0,025	34,83	181	-	-	59,99	146
0,05	35,63	273	39,97	259	60,21	268
0,075	35,29	209	40,06	172	59,99	167
0,1	34,01	246	39,44	238	60,06	193
0,15	34,26	202	39,21	225	60,24	142

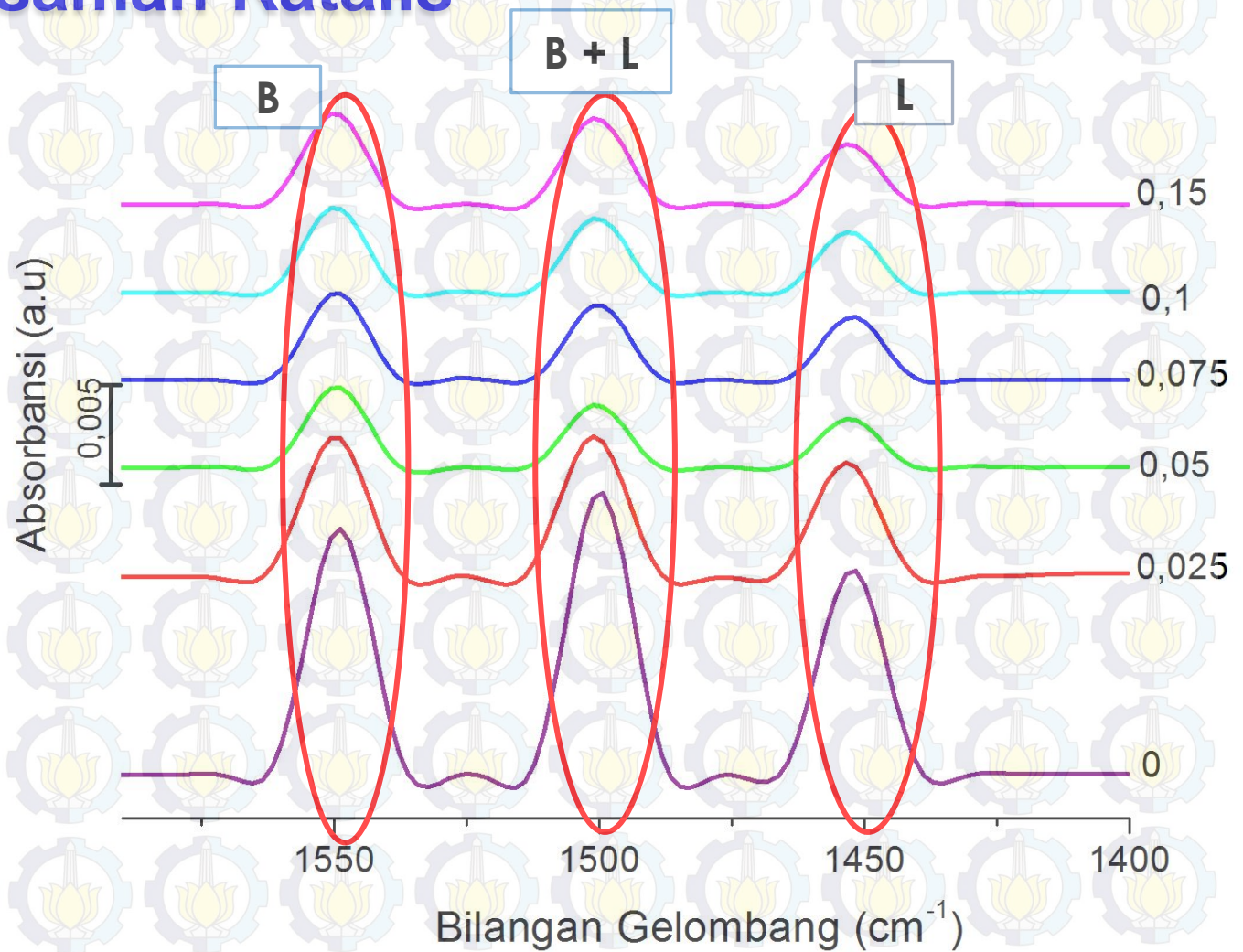
Sebelum
Kalsinasi

Setelah
Kalsinasi

FTIR



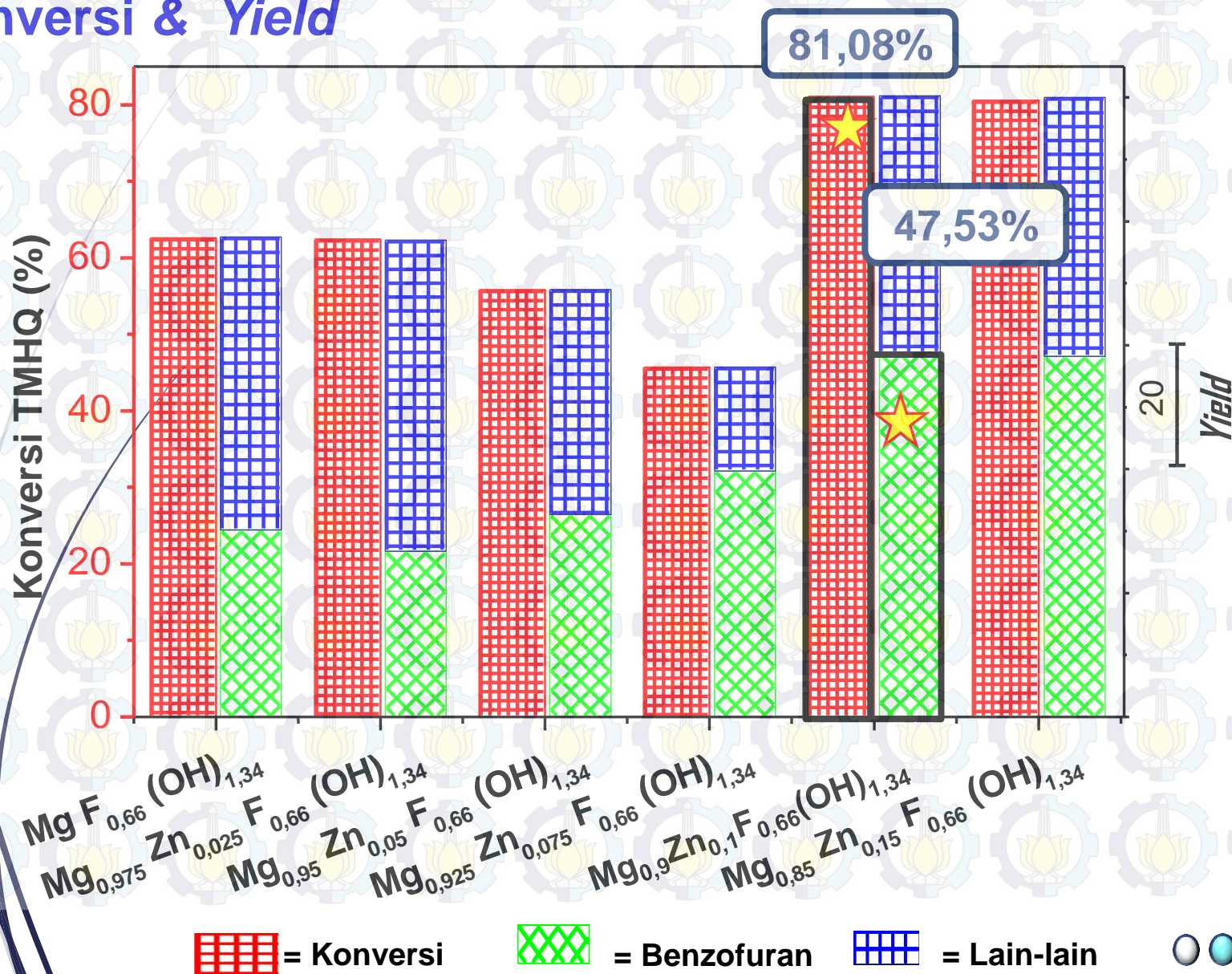
Profil Keasaman Katalis



Luas Permukaan Katalis

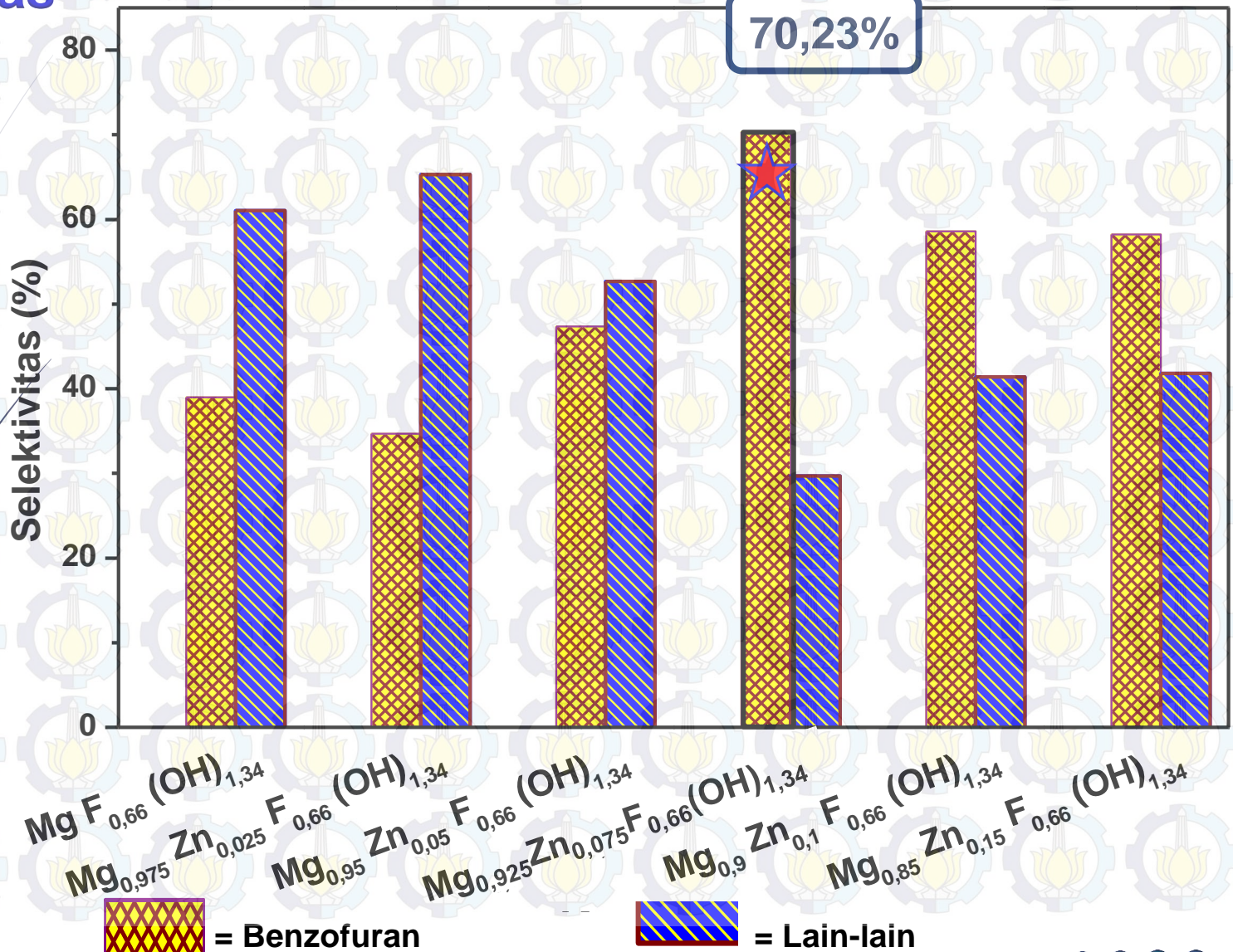
Katalis	$S_{(BET)} \text{ (m}^2\text{/g)}$
$\text{MgF}_{0,66}(\text{OH})_{1,34}$	26,884
$\text{Mg}_{0,975}\text{Zn}_{0,025}\text{F}_{0,66}(\text{OH})_{1,34}$	202,757
$\text{Mg}_{0,95}\text{Zn}_{0,05}\text{F}_{0,66}(\text{OH})_{1,34}$	369,603
$\text{Mg}_{0,925}\text{Zn}_{0,075}\text{F}_{0,66}(\text{OH})_{1,34}$	308,079
$\text{Mg}_{0,9}\text{Zn}_{0,1}\text{F}_{0,66}(\text{OH})_{1,34}$	253,720
$\text{Mg}_{0,85}\text{Zn}_{0,15}\text{F}_{0,66}(\text{OH})_{1,34}$	236,325

Konversi & Yield

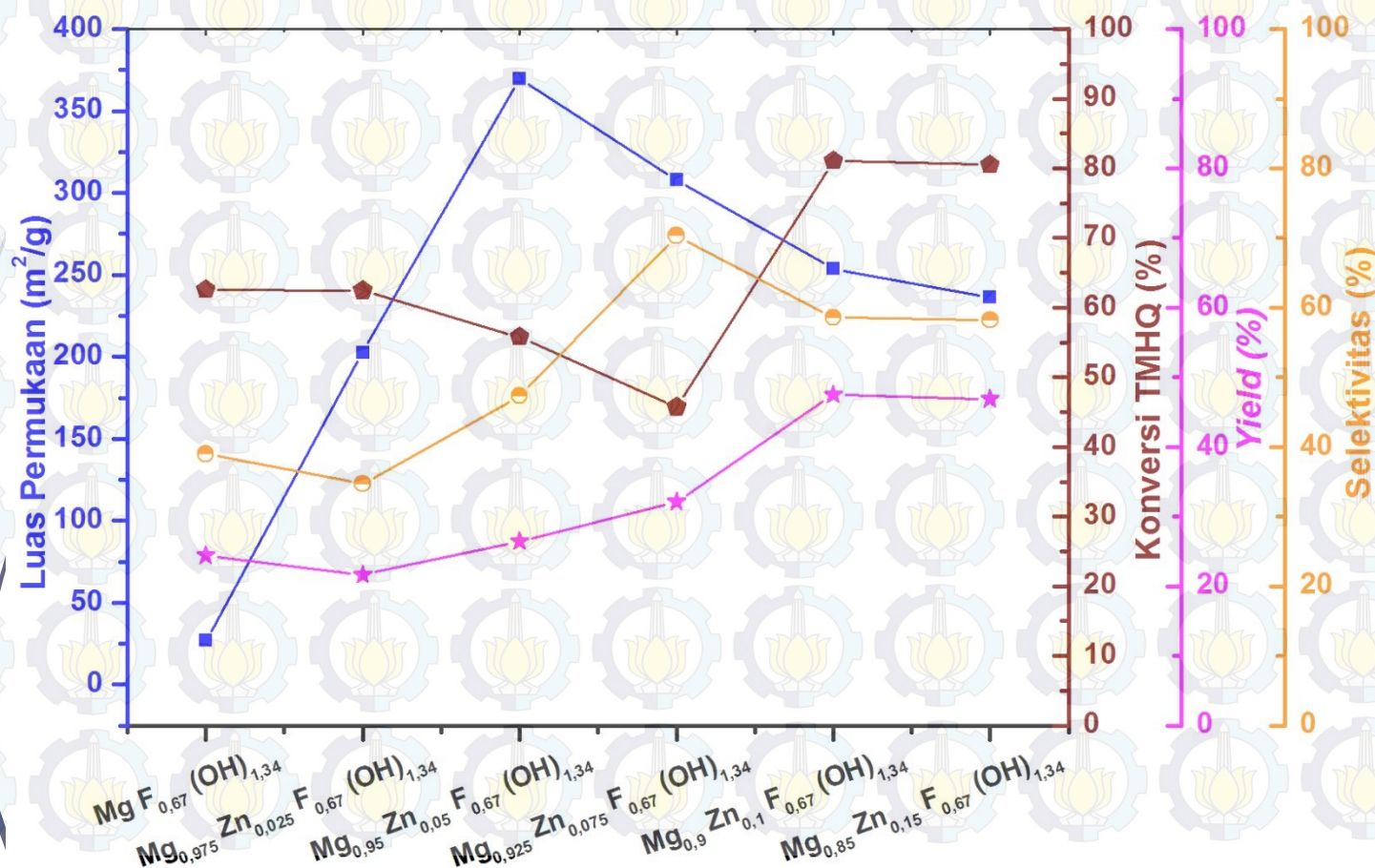


3 HASIL DAN PEMBAHASAN

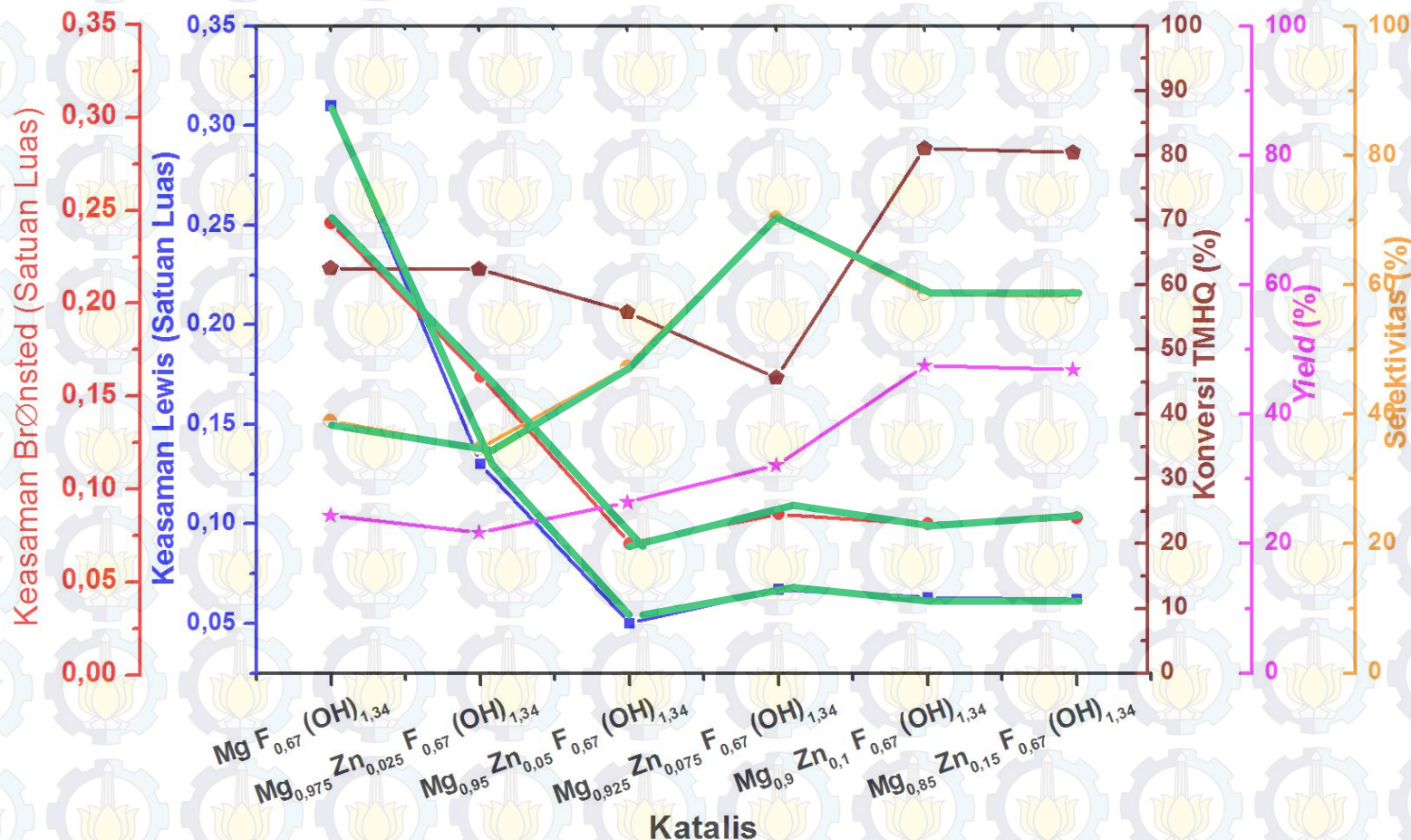
Selektivitas



Korelasi antara luas permukaan pada katalis dengan konversi TMHQ, *yield* dan selektivitas benzofuran



Korelasi antara keasaman katalis dengan konversi TMHQ, *yield* dan selektivitas benzofuran



KESIMPULAN



X= 0

X= 0,025

X= 0,05

X= 0,075

X= 0,1

X= 0,15

Konversi TMHQ

Yield

Selektivitas

81,08%

47,53 %

Benzofuran

Benzofuran

70,32%

Keasaman Katalis

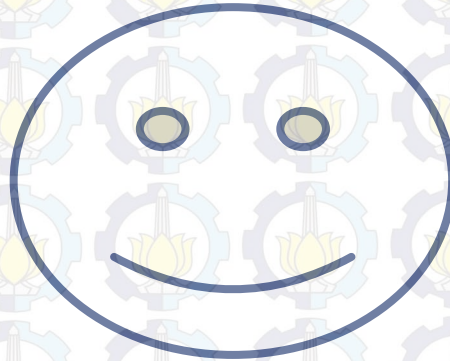
Luas Permukaan

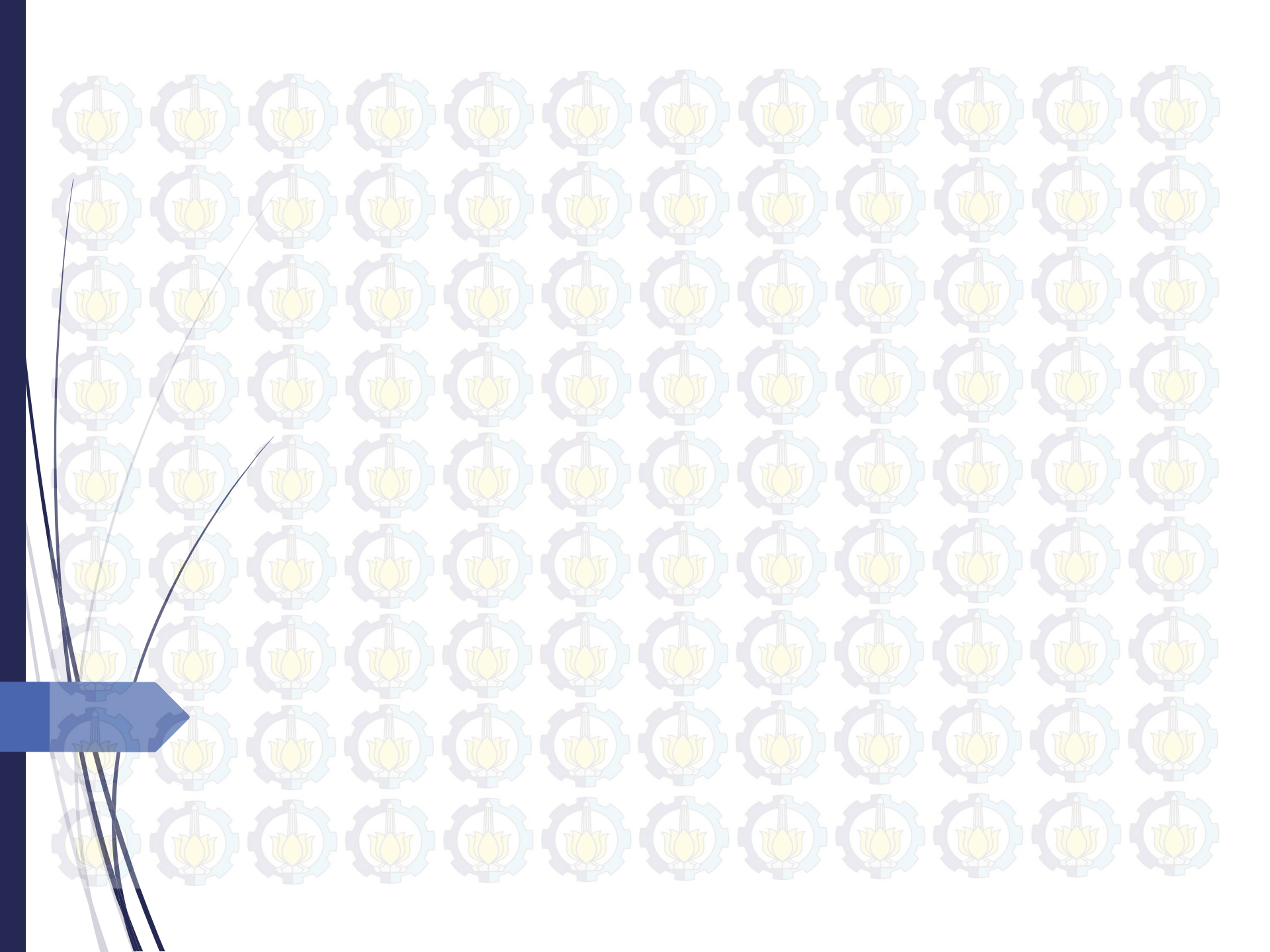
TERIMA KASIH

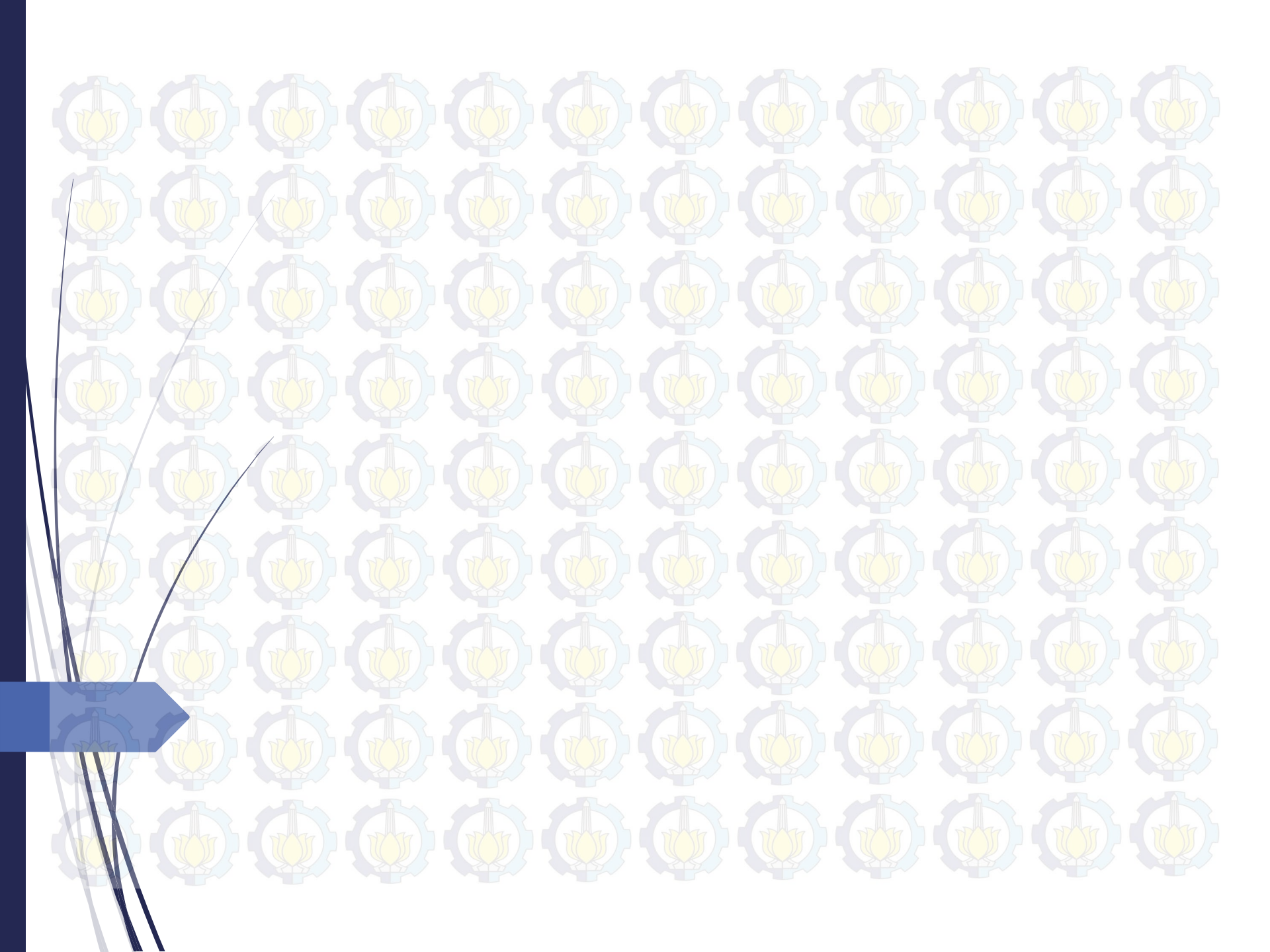
- ✓ Prof. Dr. rer. nat. Irminda Kris Murwani
- ✓ Tim Dosen Penguji
- ✓ Tim Penelitian Katalis Heterogen
- ✓ Semua pihak yang telah mendukung dalam penyusunan Tugas Akhir ini

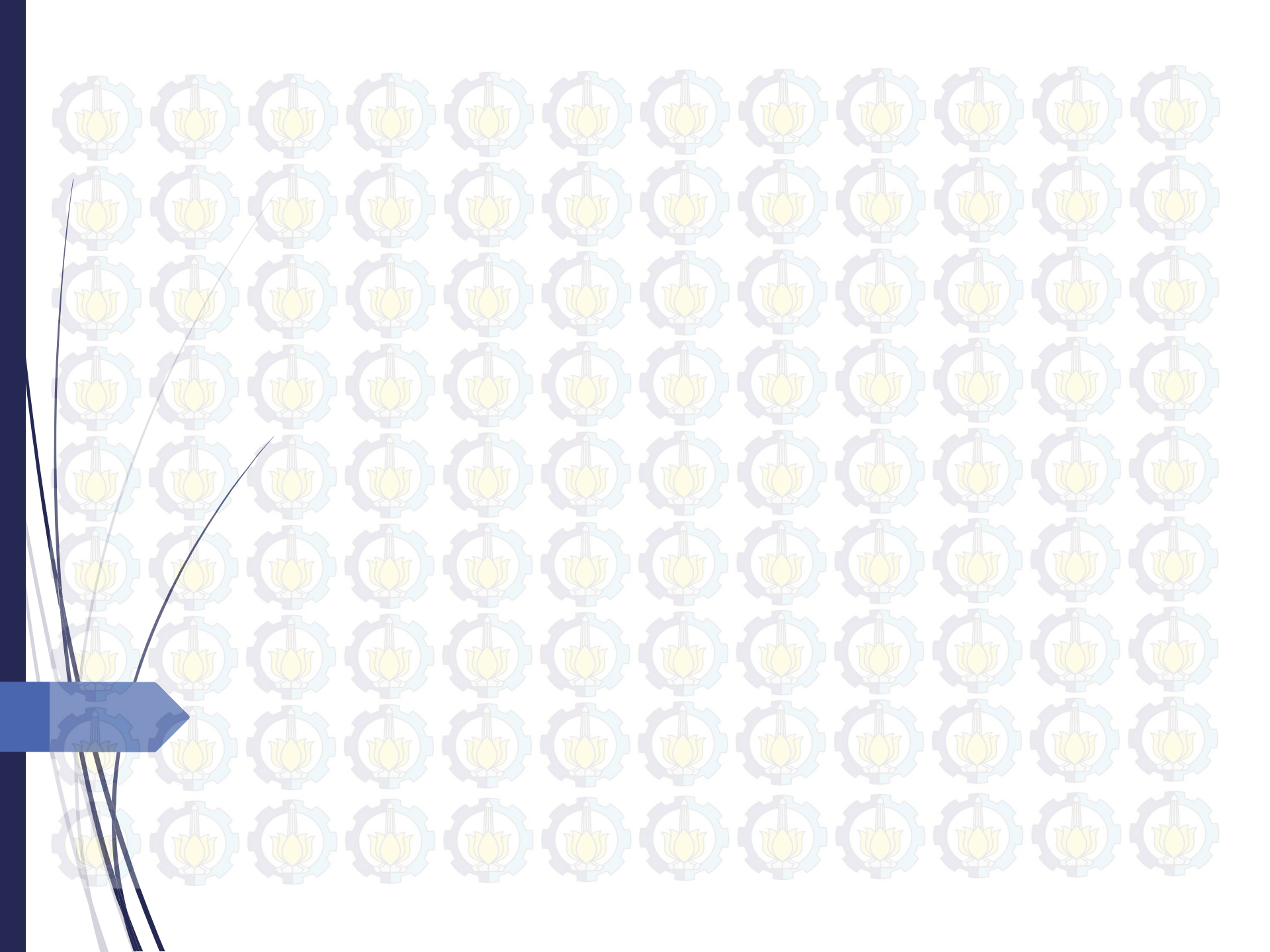


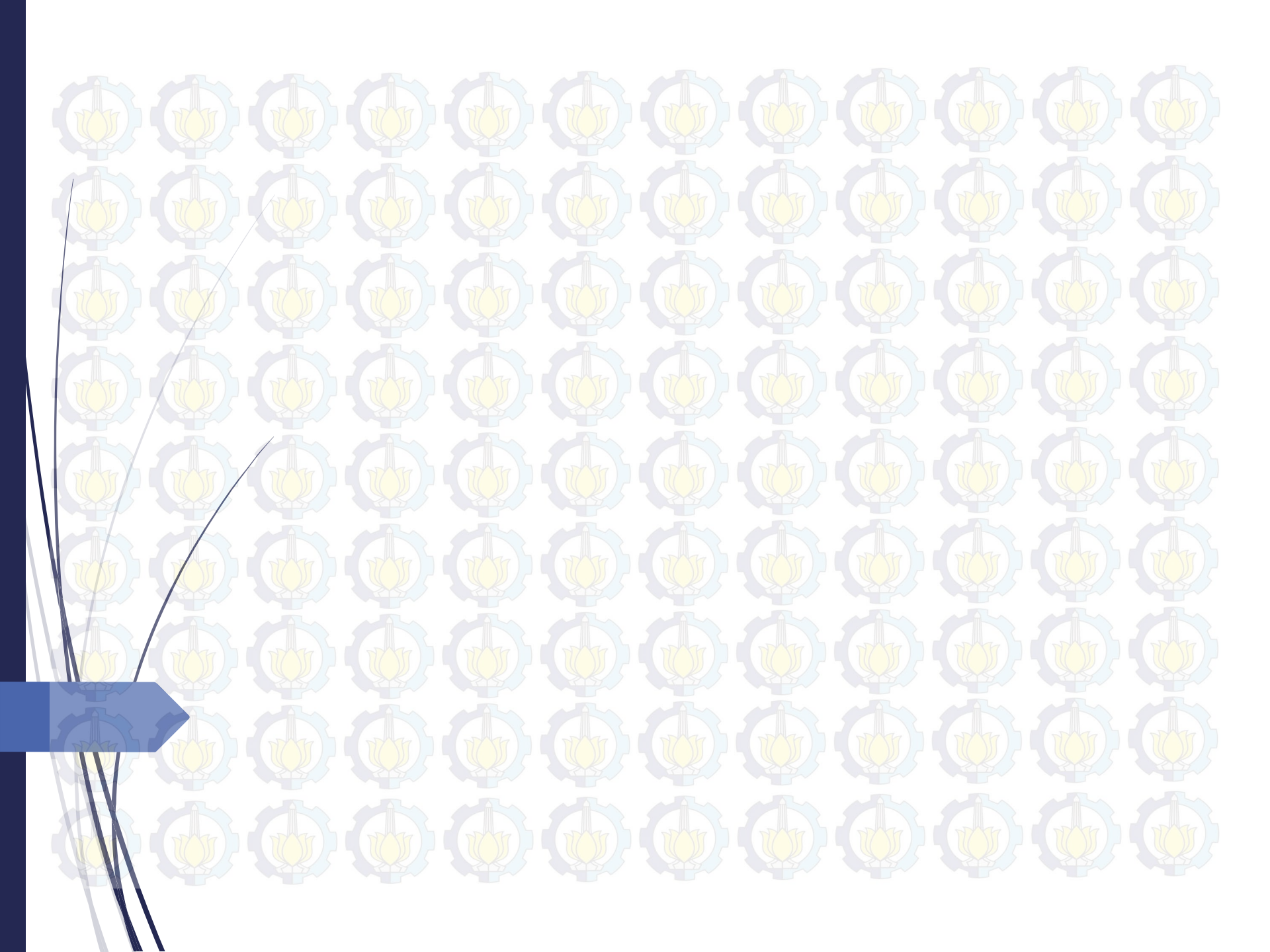
Terima Kasih

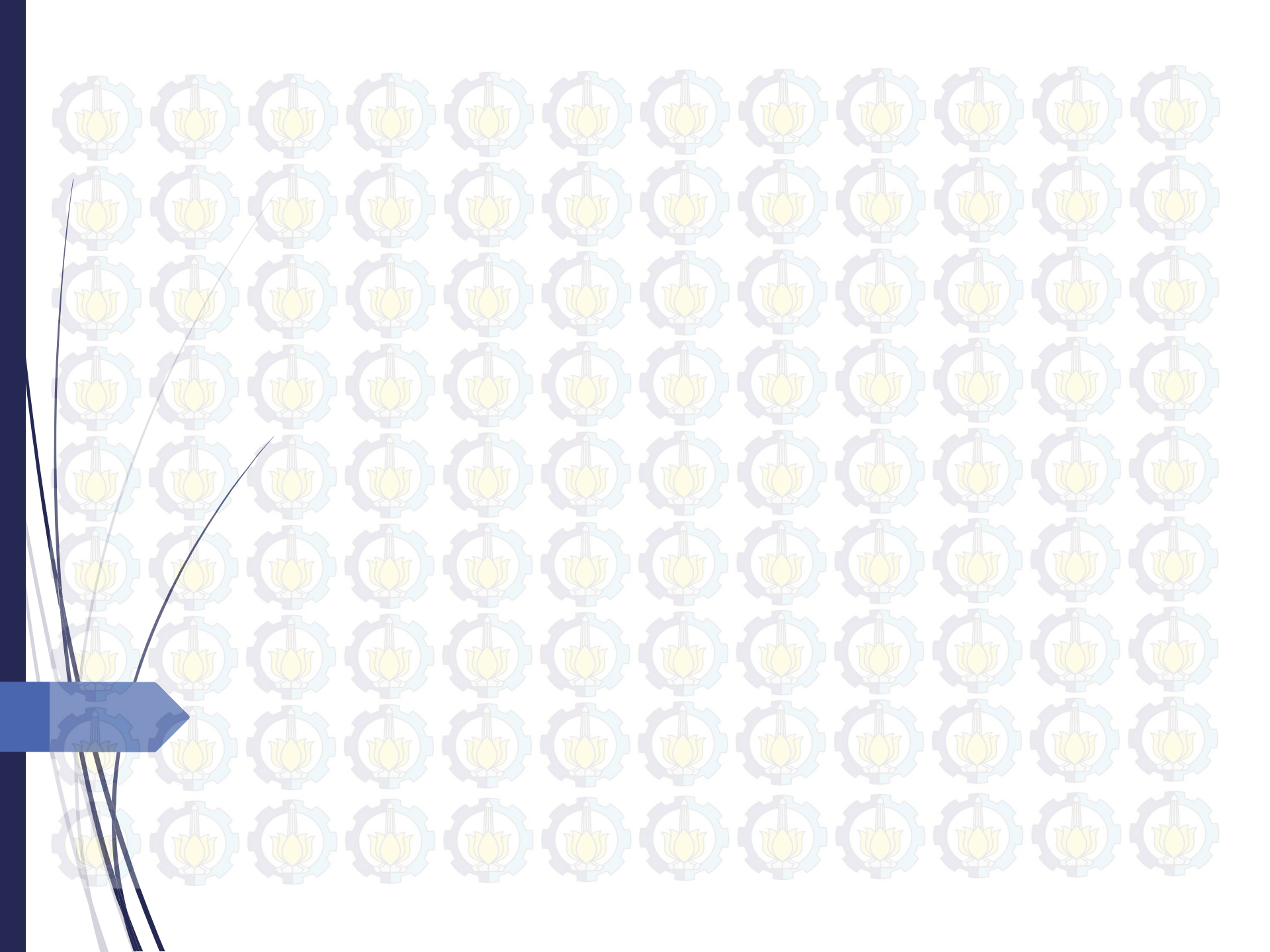


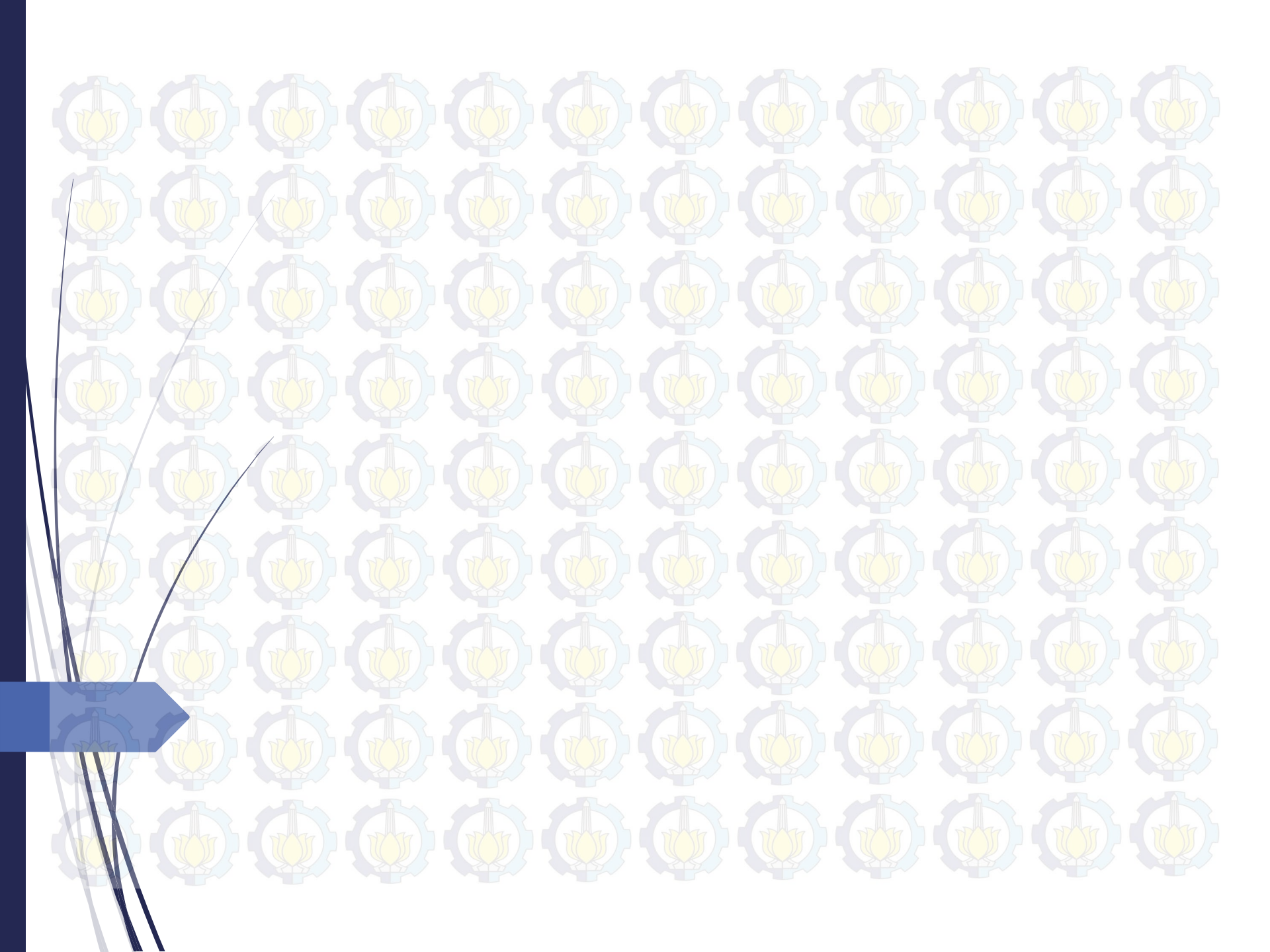


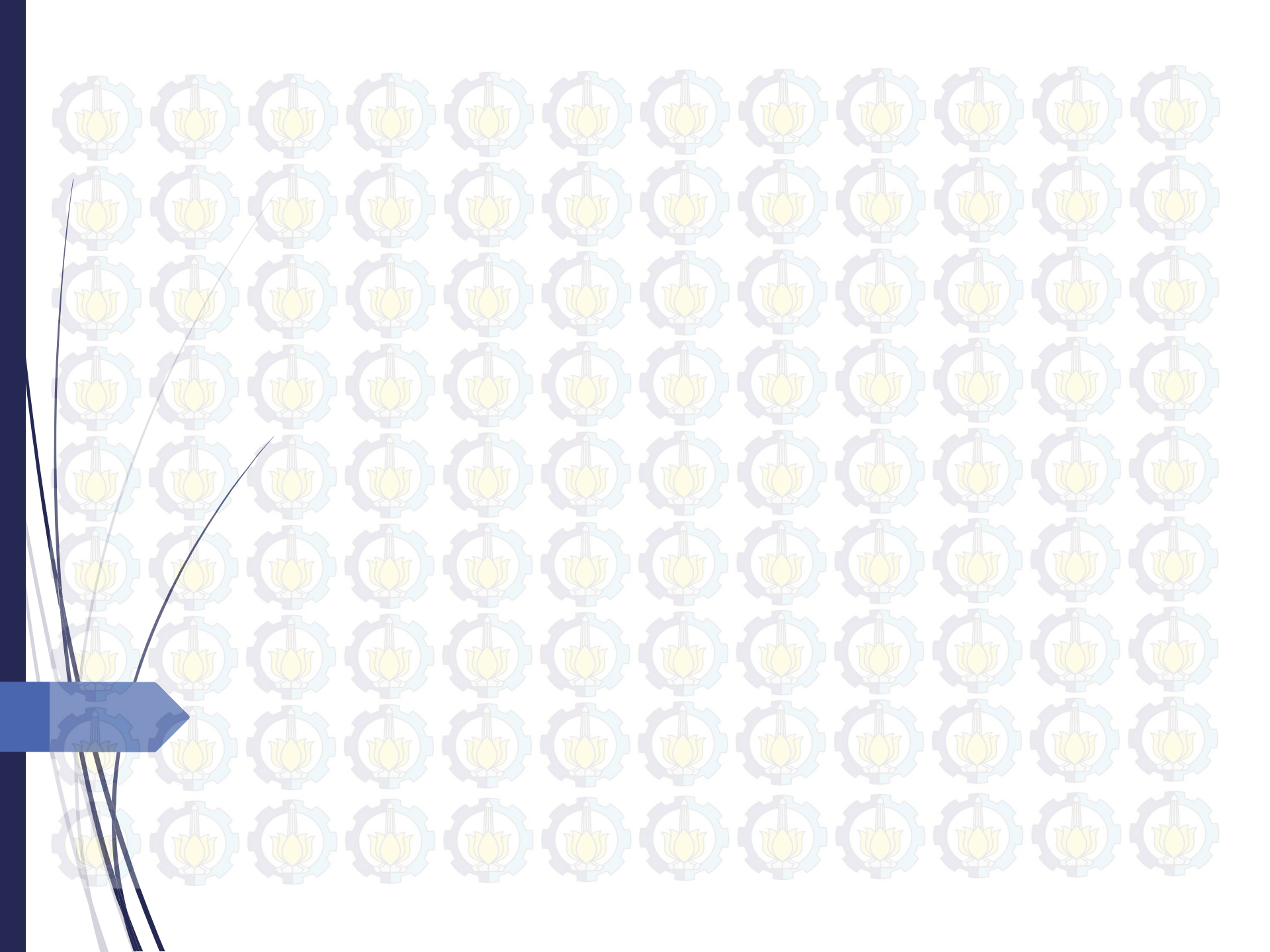


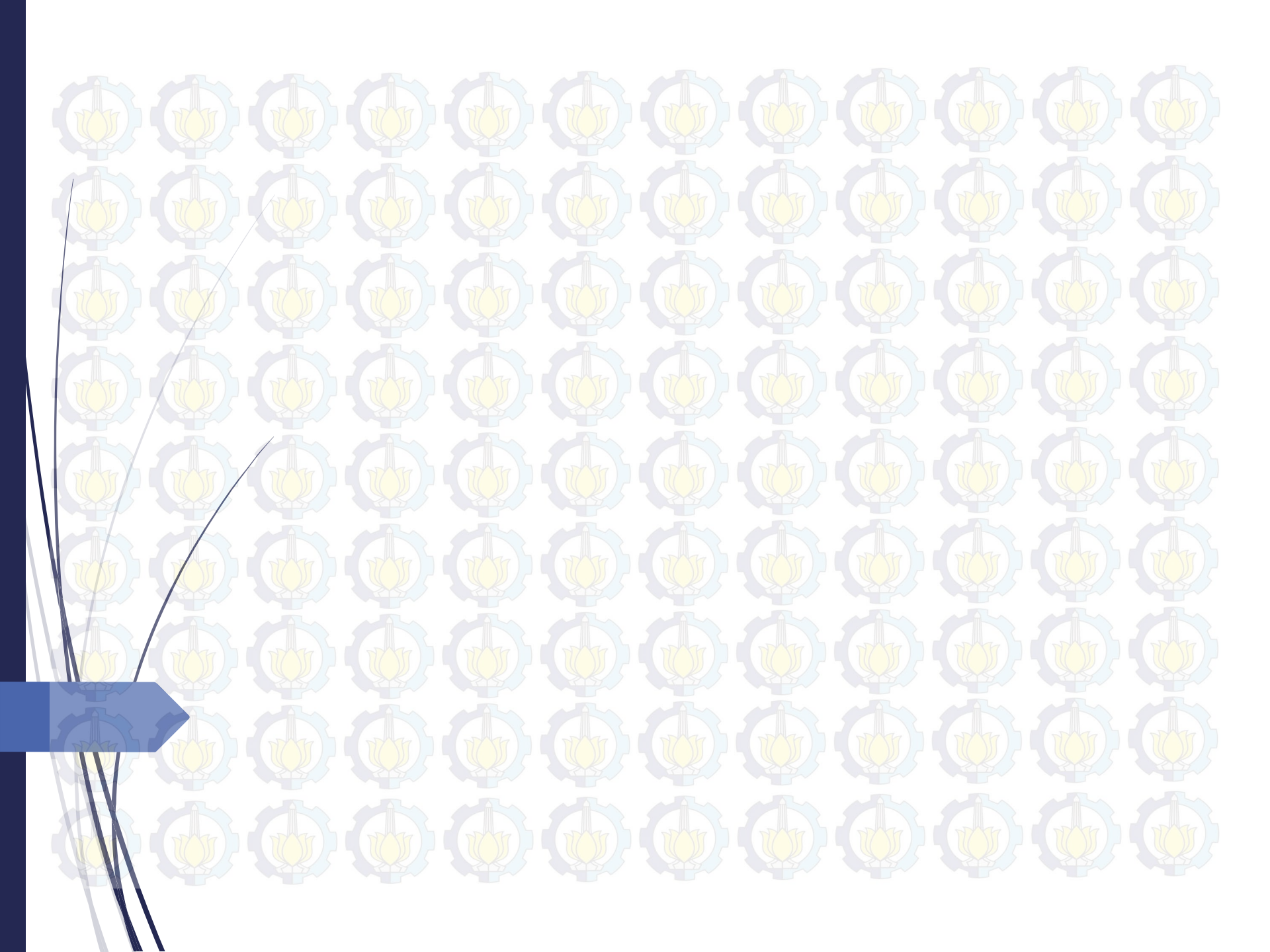












UV1100 Spectrophotometer

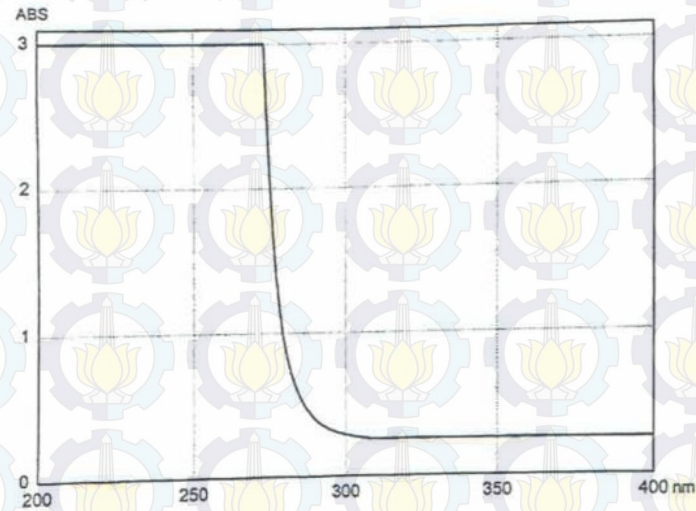
Serial NUM: 5210027

ROM Version: 20

Sample Name:

Date:

Operator:



Wavelength Scan

Data Mode:

Scan Range:

Slit Width:

Speed (nm/min) :

Lamp Change Wavelength:

Path Length:

ABS

400.0-200.0nm

4nm

200nm/min

340.0nm

Peak

WL (nm)

ABS

WL (nm)

ABS

UV1100 Spectrophotometer

Serial NUM: 5210027

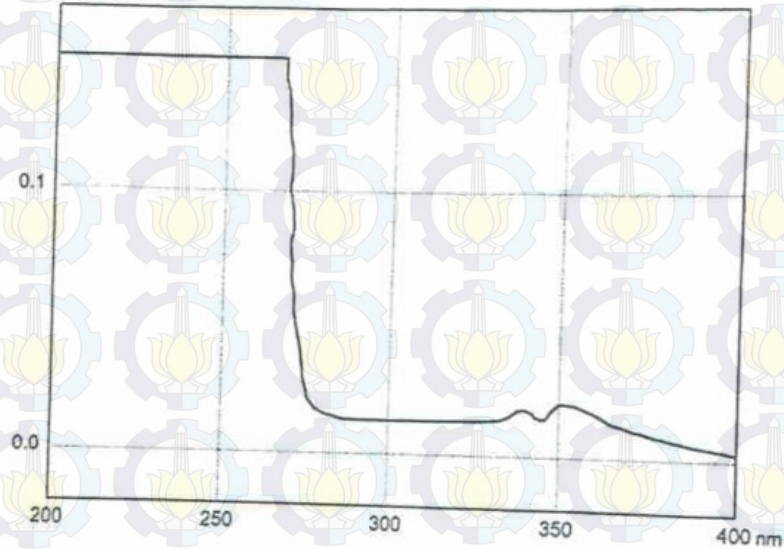
ROM Version: 20

Sample Name:

Date:

Operator:

ABS



Wavelength Scan

Data Mode:

Scan Range:

Slit Width:

Speed (nm/min) :

Lamp Change Wavelength:

Path Length:

ABS

400.0-200.0nm

4nm

200nm/min

340.0nm

Peak

WL (nm)

338.0

ABS

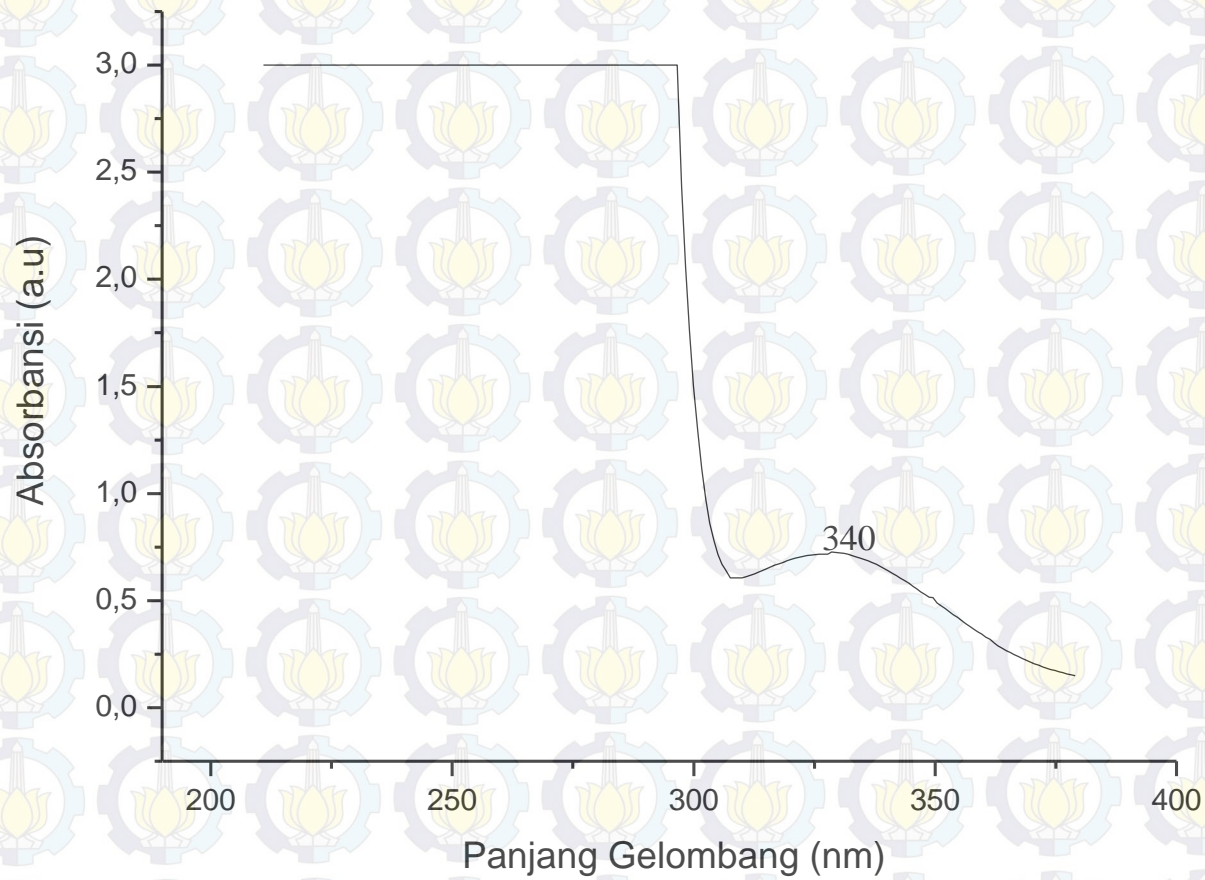
0.0165

WL (nm)

352.8

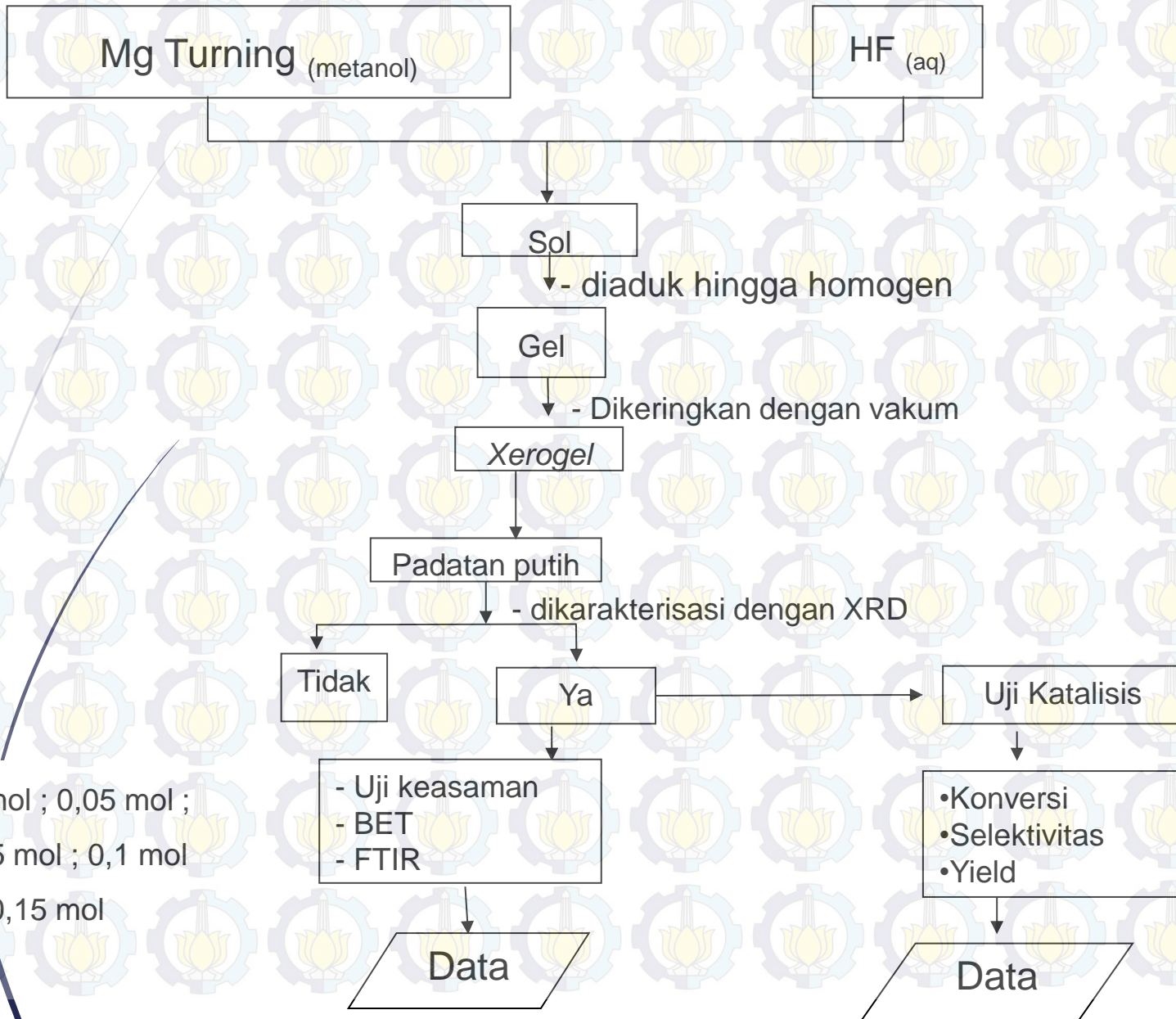
ABS

0.0253



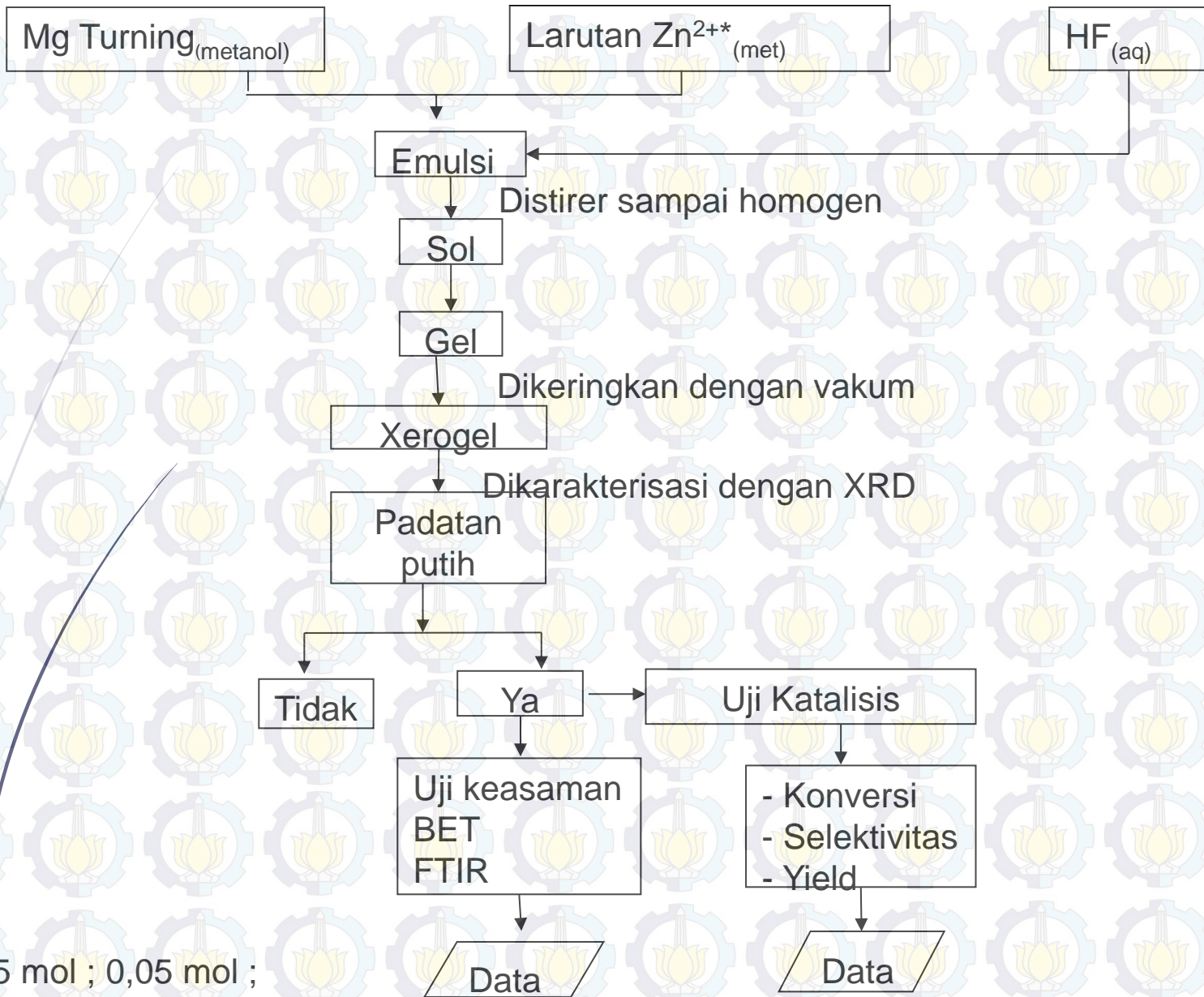
Katalis	Absorbansi
$\text{Mg}_{0,975}\text{Zn}_{0,025}\text{F}_{0,66}(\text{OH})_{1,34}$	0,025
$\text{Mg}_{0,95}\text{Zn}_{0,05}\text{F}_{0,66}(\text{OH})_{1,34}$	0,029
$\text{Mg}_{0,925}\text{Zn}_{0,075}\text{F}_{0,66}(\text{OH})_{1,34}$	0,037
$\text{Mg}_{0,9}\text{Zn}_{0,1}\text{F}_{0,66}(\text{OH})_{1,34}$	0,011
$\text{Mg}_{0,85}\text{Zn}_{0,15}\text{F}_{0,66}(\text{OH})_{1,34}$	0,012

Sintesis $\text{MgF}_{0,66}(\text{OH})_{1,34}$



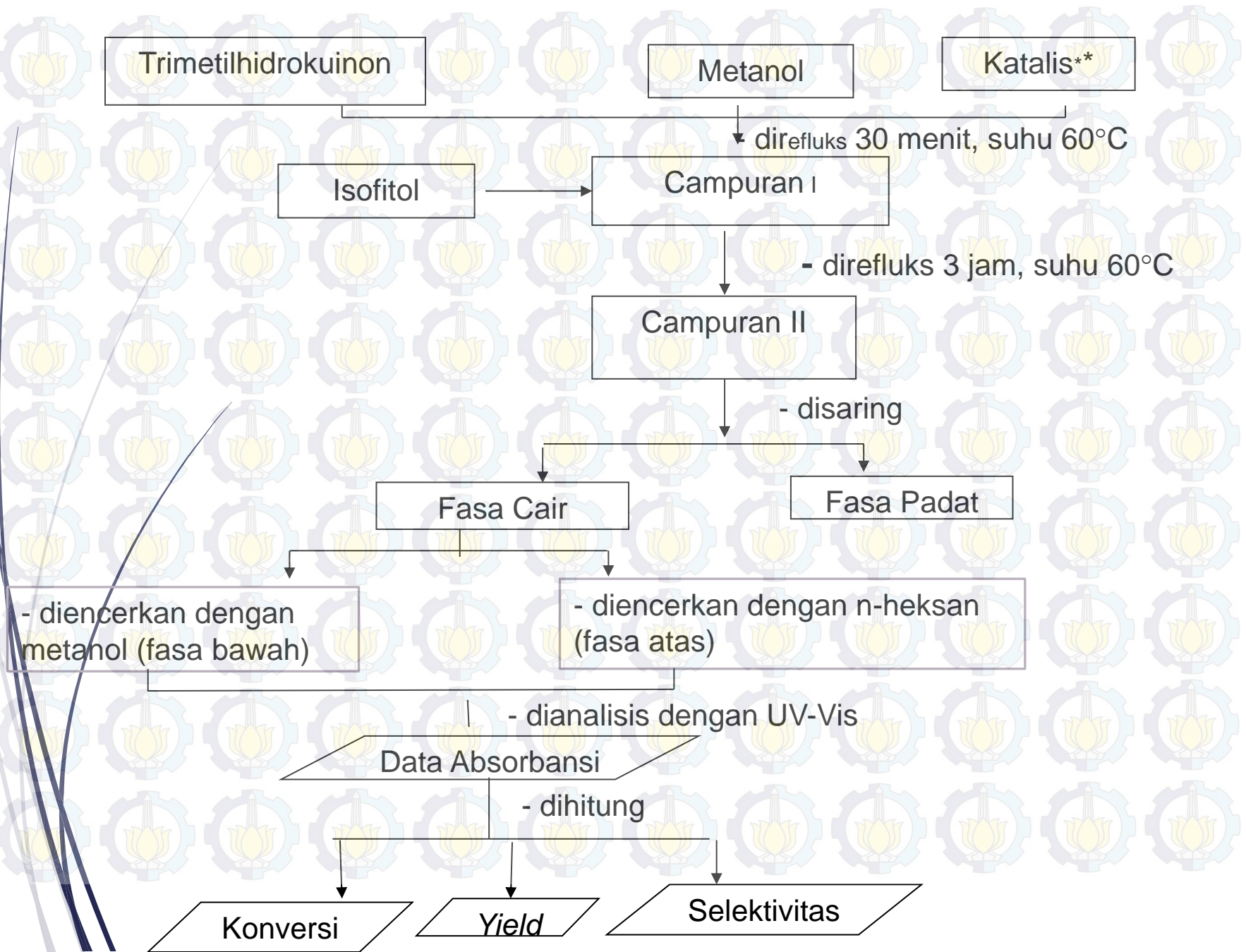
0,025 mol ; 0,05 mol ;
0,075 mol ; 0,1 mol
dan 0,15 mol

Sintesis Katalis $\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$



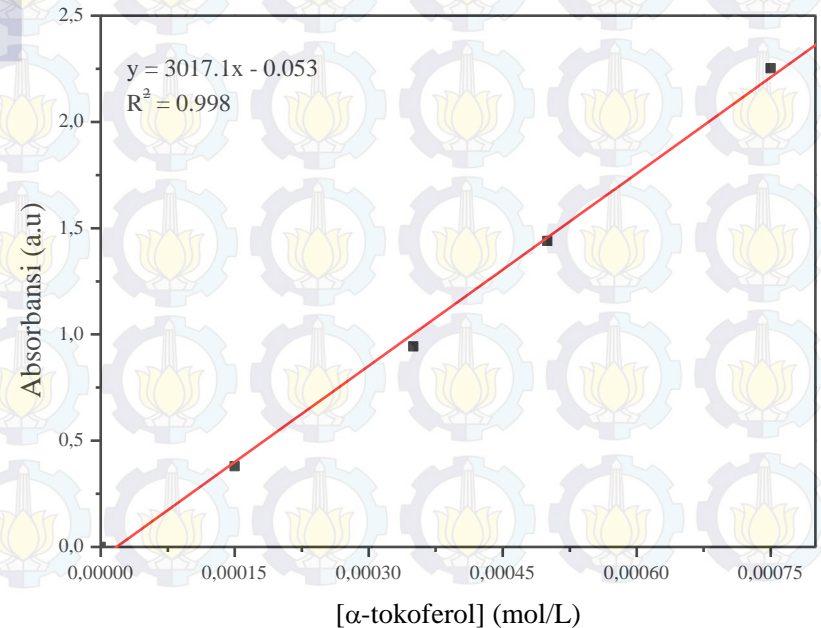
• 0,025 mol ; 0,05 mol ;
0,075 mol ; 0,1 mol
dan 0,15 mol

Uji Katalisis pada Reaksi antara Trimetilhidrokuinon dan Isofitol



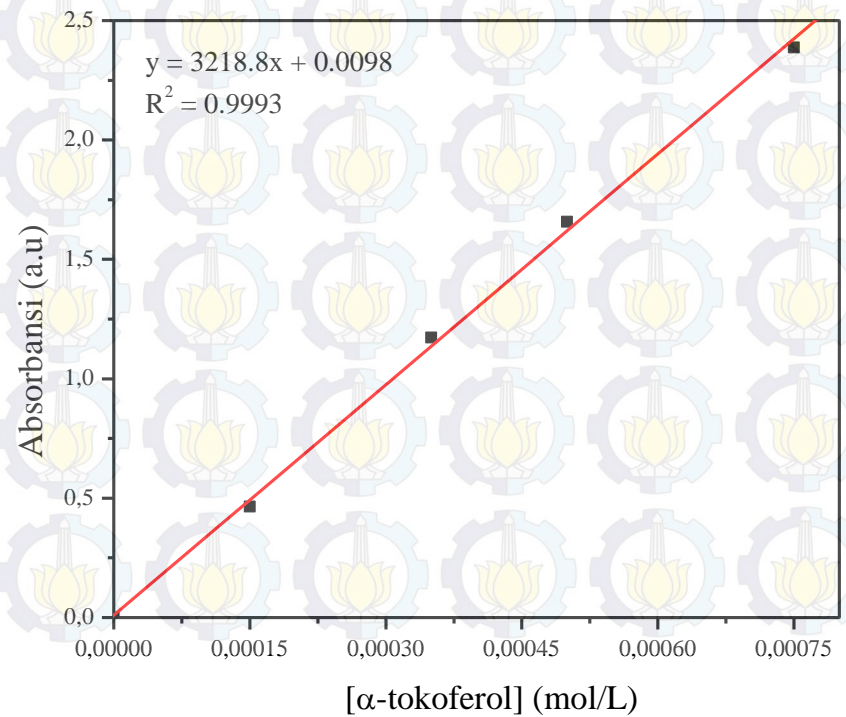
•Kurva Kalibrasi α -tokoferol dalam Metanol

Konsentrasi α -tokoferol (mol/L)	Absorbansi (a.u)
0	0
0,00016	0,00871
0,00048	0,03114
0,0008	0,05776
0,00112	0,078



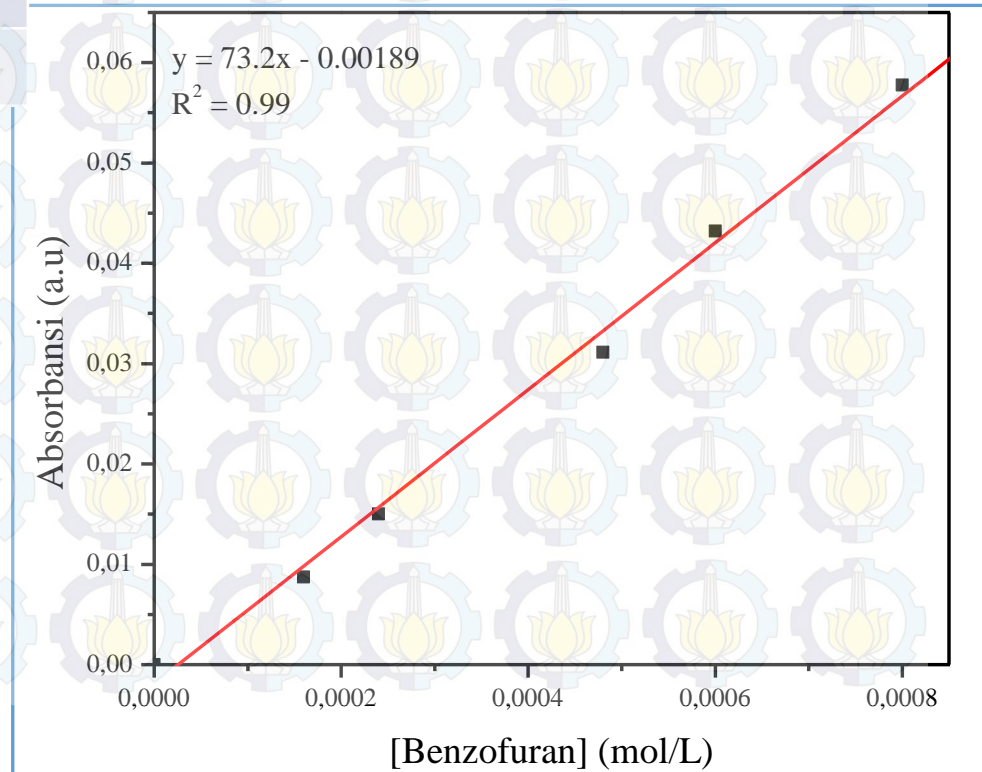
•Kurva Kalibrasi α -tokoferol dalam n-heksana

Konsentrasi α -tokoferol (mol/L)	Absorbansi (a.u)
0	0
0,00015	0,465
0,00035	1,172
0,0005	1,658
0,00075	2,387



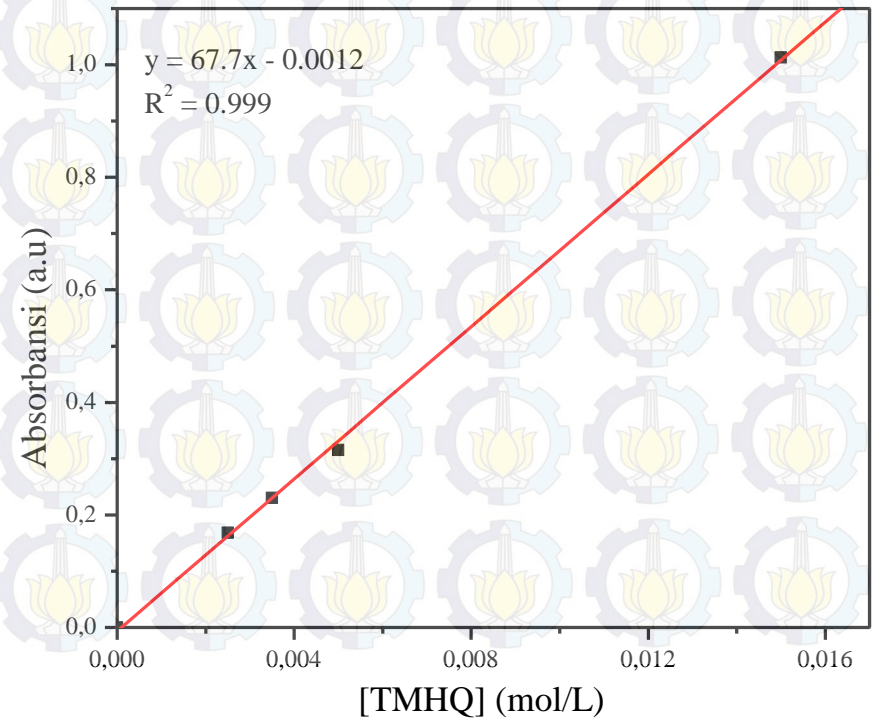
•Kurva Kalibrasi Benzofuran dalam Metanol

Konsentrasi Benzofuran (mol/L)	Absorbansi (a.u)
0	0
0,00016	0,00871
0,00024	0,015
0,00048	0,03114
0,0006	0,0432
0,0008	0,05776



•Kurva Kalibrasi TMHQ dalam Metanol

Konsentrasi TMHQ (mol/L)	Absorbansi (a.u)
0	0
0,0025	0,168
0,0035	0,23
0,005	0,315
0,015	1,013



Pengukuran absorbansi TMHQ pada $\lambda = 352,8 \text{ nm}$ adalah 0,025

$V = 10 \text{ mL} = 0,01 \text{ L}$

TMHQ_{awal}

Persamaan regresi linear TMHQ yang diperoleh :

$$y = 67,7x - 0,0012$$

dimana :

$$y = A + Bx$$

dengan, $x = \text{Konsentrasi (mol/L)}$

$y = \text{Absorbansi}$

$$\text{mol TMHQ sisa} = 0,000387 \text{ mol/L} \times 0,01 \text{ L} \times 10$$

$$= 0,0000387 \text{ mol}$$

$$= 0,0387 \text{ mmol}$$

Konversi atau Aktivitas

Yield dan Selektivitas

Pengukuran absorbansi benzofuran pada $\lambda = 338 \text{ nm}$ adalah 0,0142.

$$V = 10 \text{ mL} = 0,01 \text{ L}$$

Persamaan regresi linear α -tokoferol yang diperoleh :

$$y = 73,2x - 0,00189$$

dimana :

$$y = A + Bx$$

dengan, $x = \text{Konsentrasi (mol/L)}$

$y = \text{Absorbansi}$

$$\text{mol benzofuran} = 0,000219 \text{ mol/L} \times 0,01 \text{ L} \times 10$$

$$= 0,0000219 \text{ mol}$$

$$= 0,0219 \text{ mmol}$$

Yield

$$= 21,89 \%$$

Selektivitas

Sebelum kalsinasi



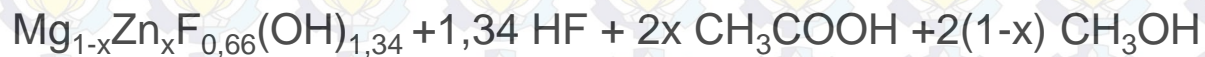
Setelah kalsinasi



Katalis	Konversi (%)	Yield (%)	
		Benzofuran	Lain-lain
$\text{MgF}_{0,67}(\text{OH})_{1,34}$	62,582440	24,389828	38,192612
$\text{Mg}_{0,975}\text{Zn}_{0,025}\text{F}_{0,67}(\text{OH})_{1,3}$ 4	62,443283	21,666088	40,777195
$\text{Mg}_{0,95}\text{Zn}_{0,05}\text{F}_{0,67}(\text{OH})_{1,34}$	55,802965	26,417057	29,385908
$\text{Mg}_{0,925}\text{Zn}_{0,075}\text{F}_{0,67}(\text{OH})_{1,3}$ 4	45,693346	32,132467	13,560878
$\text{Mg}_{0,9}\text{Zn}_{0,1}\text{F}_{0,67}(\text{OH})_{1,34}$	81,079821	47,525754	33,554067
$\text{Mg}_{0,85}\text{Zn}_{0,15}\text{F}_{0,67}(\text{OH})_{1,34}$	80,533304	46,876282	33,657022

Katalis	Selektivitas (%)	
	Benzofuran	Lain-Lain
$\text{MgF}_{0,67}(\text{OH})_{1,34}$	38,972319	61,027681
$\text{Mg}_{0,975}\text{Zn}_{0,025}\text{F}_{0,67}(\text{OH})_{1,34}$	34,697227	65,302773
$\text{Mg}_{0,95}\text{Zn}_{0,05}\text{F}_{0,67}(\text{OH})_{1,34}$	47,339881	52,660119
$\text{Mg}_{0,925}\text{Zn}_{0,075}\text{F}_{0,67}(\text{OH})_{1,34}$	70,321984	29,678016
$\text{Mg}_{0,9}\text{Zn}_{0,1}\text{F}_{0,67}(\text{OH})_{1,34}$	58,616007	41,383993
$\text{Mg}_{0,85}\text{Zn}_{0,15}\text{F}_{0,67}(\text{OH})_{1,34}$	58,207325	41,792675

Perhitungan Sintesis $\text{Mg}_{1-x}\text{Zn}_x\text{F}_{0,66}(\text{OH})_{1,34}$



❑ Massa senyawa target = 2 g

❖ Menghitung Mr produk

$$\text{Mg} = 0,975 \times 24,38 = 23,4$$

$$\text{Zn} = 0,025 \times 65,37 = 1,63$$

$$\text{F} = 0,66 \times 19 = 12,54$$

$$\text{OH} = 1,34 \times 17 = 22,78$$

$$= 60,35 \text{ g/mol}$$

$$\text{Mol Produk} = \frac{2}{60,35} = 0,033 \text{ mol}$$

$$\text{Kebutuhan Mg Turning} = 0,975 \times 0,033 \times 24,3 = 0,782 \text{ g}$$

$$\text{Kebutuhan HF} = \frac{0,033}{27,12} \times 0,67 \times 1000 = 0,82 \text{ mL}$$

$$\text{Kebutuhan Logam Zn} = 0,025 \times 0,033 \times 219,49 = 0,1811$$

TABEL PERIODIK UNSUR KIMIA

GOLONGAN

1 1.00797 H 1s ¹ Hidrogen																		2 4.0026 He 1s ² Helium	
IIA																			
3 6.939 Li 1s ² 2s ¹ Litium	4 9.0122 Be 1s ² 2s ² Berilium																	10 20.183 Ne 1s ² 2s ² 2p ⁶ Neon	
11 22.9898 Na [Ne] 3s ¹ Natrium	12 24.312 Mg [Ne] 3s ² Magnesium																	18 39.948 Ar [Ne] 3s ² 3p ⁶ Argon	
IIIB		IVB		VB		VIB		VIIB		VIIIB		IIIB		IIB		IB		IIB	
19 39.102 K [Ar] 4s ¹ Kalium	20 40.08 Ca [Ar] 4s ² Kalsium	21 44.956 Sc [Ar] 3d ¹ 4s ² Skandium	22 47.90 Ti [Ar] 3d ² 4s ² Titan	23 50.942 V [Ar] 3d ³ 4s ² Vanadium	24 51.996 Cr [Ar] 3d ⁵ 4s ¹ Krom	25 54.938 Mn [Ar] 3d ⁵ 4s ² Mangan	26 55.847 Fe [Ar] 3d ⁶ 4s ² Besi	27 58.933 Co [Ar] 3d ⁷ 4s ² Kobalt	28 58.71 Ni [Ar] 3d ⁸ 4s ² Nikel	29 63.546 Cu [Ar] 3d ¹⁰ 4s ¹ Tembaga	30 65.37 Zn [Ar] 3d ¹⁰ 4s ² Seng	31 69.72 Ga [Ar] 3d ¹⁰ 4s ² 4p ¹ Gallium	32 72.59 Ge [Ar] 3d ¹⁰ 4s ² 4p ² Germanium	33 74.922 As [Ar] 3d ¹⁰ 4s ² 4p ³ Arsen	34 78.96 Se [Ar] 3d ¹⁰ 4s ² 4p ⁴ Selen	35 79.904 Br [Ar] 3d ¹⁰ 4s ² 4p ⁵ Brom	36 83.80 Kr [Ar] 3d ¹⁰ 4s ² 4p ⁶ Krypton		
37 85.47 Rb [Kr] 5s ¹ Rubidium	38 87.62 Sr [Kr] 5s ² Strontium	39 88.906 Y [Kr] 4d ¹ 5s ² Litium	40 91.22 Zr [Kr] 4d ² 5s ² Sirkon	41 92.906 Nb [Kr] 4d ⁴ 5s ¹ Niobium	42 95.94 Mo [Kr] 4d ⁵ 5s ¹ Molibden	43 98.906 Tc [Kr] 4d ⁵ 5s ² Teknesium	44 101.07 Ru [Kr] 4d ⁷ 5s ¹ Rutenium	45 102.908 Rh [Kr] 4d ⁸ 5s ¹ Rodium	46 106.4 Pd [Kr] 4d ¹⁰ 5s ⁰ Paladium	47 107.87 Ag [Kr] 4d ¹⁰ 5s ¹ Perak	48 112.40 Cd [Kr] 4d ¹⁰ 5s ² Kadmium	49 114.82 In [Kr] 4d ¹⁰ 5s ² 5p ² Indium	50 118.6 Sn [Kr] 4d ¹⁰ 5s ² 5p ² Timah	51 121.75 Sb [Kr] 4d ¹⁰ 5s ² 5p ³ Antimon	52 127.60 Te [Kr] 4d ¹⁰ 5s ² 5p ⁴ Telurium	53 126.904 I [Kr] 4d ¹⁰ 5s ² 5p ⁵ Yod	54 131.30 Xe [Kr] 4d ¹⁰ 5s ² 5p ⁶ Xenon		
55 132.905 Cs [Xe] 6s ¹ Sesium	56 137.34 Ba [Xe] 6s ² Barium	57 138.91 La [Xe] 5d ¹ 6s ² Lantan	58 140.91 Ce [Xe] 4f ¹ 5d ¹ 6s ² Lantan	59 140.907 Pr [Xe] 4f ³ 6s ² Praseodimium	60 144.24 Nd [Xe] 4f ⁴ 6s ² Neodimium	61 147.07 Pm [Xe] 4f ⁵ 6s ² Prometium	62 150.35 Sm [Xe] 4f ⁶ 6s ² Samarium	63 151.96 Eu [Xe] 4f ⁷ 6s ² Europium	64 157.25 Gd [Xe] 4f ⁷ 5d ¹ 6s ² Gadolinium	65 158.92 Tb [Xe] 4f ⁹ 6s ² Terbium	66 162.50 Dy [Xe] 4f ¹⁰ 6s ² Dysprosium	67 164.93 Ho [Xe] 4f ¹¹ 6s ² Holmium	68 167.26 Er [Xe] 4f ¹² 6s ² Erbium	69 168.93 Tm [Xe] 4f ¹³ 6s ² Terbium	70 173.04 Yb [Xe] 4f ¹⁴ 6s ² Ytterbium	71 174.97 Lu [Xe] 4f ¹⁴ 6s ² Lutetium			
87 223.018 Fr [Rn] 7s ¹ Francium	88 226.025 Ra [Rn] 7s ² Radium	89 227.033 Ac [Rn] 6d ¹ 7s ² Aktinium	90 223.018 Th [Rn] 6d ² 7s ² Torium	91 227.033 Pa [Rn] 5f ² 6d ¹ 7s ² Protaktinium	92 238.029 U [Rn] 5f ³ 6d ¹ 7s ² Uranium	93 237.043 Np [Rn] 5f ⁴ 6d ¹ 7s ² Neptunium	94 237.043 Pu [Rn] 5f ⁶ 6d ¹ 7s ² Plutonium	95 238.029 Am [Rn] 5f ⁷ 6d ¹ 7s ² Amerisium	96 243.061 Cm [Rn] 5f ⁷ 6d ² 7s ² Kurisium	97 247.073 Bk [Rn] 5f ⁹ 6d ¹ 7s ² Berkelium	98 250.106 Cf [Rn] 5f ¹⁰ 6d ¹ 7s ² Kalifornium	99 252.083 Es [Rn] 5f ¹¹ 6d ¹ 7s ² Einstenium	100 254.089 Fm [Rn] 5f ¹² 6d ¹ 7s ² Fermium	101 256.105 Md [Rn] 5f ¹³ 6d ¹ 7s ² Mendelevium	102 259.108 No [Rn] 5f ¹⁴ 6d ¹ 7s ² Nobelium	103 262.109 Lw [Rn] 5f ¹⁴ 6d ² 7s ² Lawrensium			

Nomor atom

30

KUNCI

2

Massa Atom (2)

65.37

Tingkat oksidasi

2

Titik didih C

906

Titik leleh

419.5

Massa jenis (g/m³) (3)

7.14

Lambang (1—)

Zn

Struktur elektron

[Ar] 3d10⁴s²

S e n g

Nama

Dipakai untuk :

* SMA * SAA * SITA * Lainnya

Nomor atom → 30
KUNCI → 2
Massa Atom (2) → 65.37
Tingkat oksidasi → 2
Titik didih C → 906
Titik leleh → 419.5
Massa jenis (g/m³) (3) → 7.14
Lambang (1-) → Zn
Struktur elektron → [Ar] 3d¹⁰4s²
Nama → Seng

Dipakai untuk :

• S.M.A. — S.A.A & S.I.T.A. Lainnya

• Universitas

CATATAN WARNA

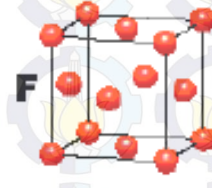
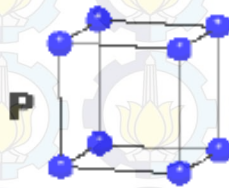
- Hijau telor = padat
Orange = gas
Kuning Tua = Cair
Merah jambu = unsur buatan
- Didasarkan atas karbon — 12
Tanda () menyatakan isotop paling stabil.
- Untuk unsur berfasa gas harga tersebut berarti titik didih cairannya.

Geometri kristal

CUBIC

$$a = b = c$$

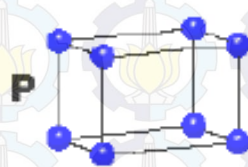
$$\alpha = \beta = \gamma = 90^\circ$$



TETRAGONAL

$$a = b \neq c$$

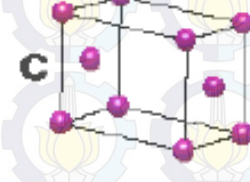
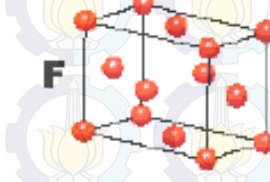
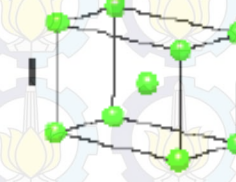
$$\alpha = \beta = \gamma = 90^\circ$$



ORTHORHOMBIC

$$a \neq b \neq c$$

$$\alpha = \beta = \gamma = 90^\circ$$



HEXAGONAL

$$a = b \neq c$$

$$\alpha = \beta = 90^\circ$$

$$\gamma = 120^\circ$$



TRIGONAL

$$a = b = c$$

$$\alpha = \beta = \gamma \neq 90^\circ$$

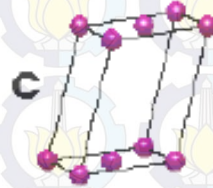


MONOCLINIC

$$a \neq b \neq c$$

$$\alpha = \gamma = 90^\circ$$

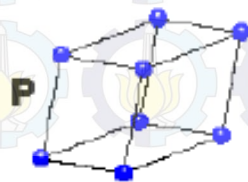
$$\beta \neq 120^\circ$$



TRICLINIC

$$a \neq b \neq c$$

$$\alpha \neq \beta \neq \gamma \neq 90^\circ$$



4 Types of Unit Cell

P = Primitive

I = Body-Centred

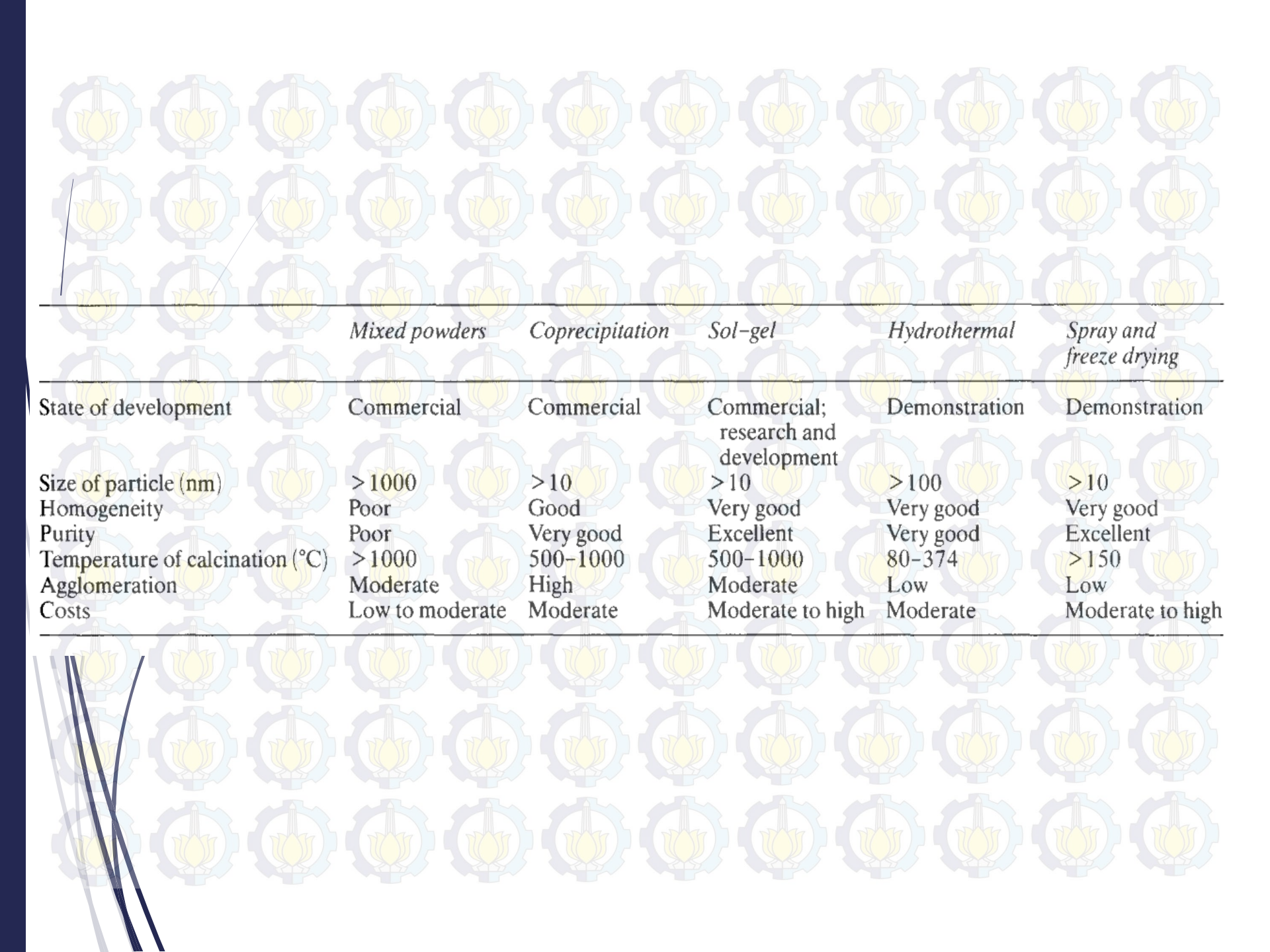
F = Face-Centred

C = Side-Centred

+

7 Crystal Classes

→ 14 Bravais Lattices



	<i>Mixed powders</i>	<i>Coprecipitation</i>	<i>Sol-gel</i>	<i>Hydrothermal</i>	<i>Spray and freeze drying</i>
State of development	Commercial	Commercial	Commercial; research and development	Demonstration	Demonstration
Size of particle (nm)	> 1000	> 10	> 10	> 100	> 10
Homogeneity	Poor	Good	Very good	Very good	Very good
Purity	Poor	Very good	Excellent	Very good	Excellent
Temperature of calcination (°C)	> 1000	500–1000	500–1000	80–374	> 150
Agglomeration	Moderate	High	Moderate	Low	Low
Costs	Low to moderate	Moderate	Moderate to high	Moderate	Moderate to high